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PAPER

# $Fe_3O_4$@$SiO_2$ nanoparticles as high-performance Fenton-like catalyst in neutral environment

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Advanced oxidation processes (AOP) have been widely applied in water treatment. However, traditional Fenton reaction based on $Fe^{2+}$-$H_2O_2$ system requires acidic environment and generates large amount of $Fe^{3+}$ ions. Herein, we reported that magnetic $Fe_3O_4$ core-$SiO_2$ shell nanoparticles ($Fe_3O_4$@$SiO_2$ NPs) could be used as Fenton-like catalyst for the decomposition of $H_2O_2$, resulting in the decoloration of methylene blue (MB). $Fe_3O_4$@$SiO_2$ NPs had much higher activity than bare $Fe_3O_4$ cores, suggesting the coating of $SiO_2$ enhanced the catalytic activity. Most importantly, the best performance of $Fe_3O_4$@$SiO_2$ NPs was observed at neutral pH values. Higher temperature facilitated the diffusion of MB in solution, thus, promoted the decoloration efficiency. The radical reaction nature was reflected by the electron spin resonance spectrum and the significant inhibition of the decoloration in the presence of radical scavenger tertiary butanol. $Fe_3O_4$@$SiO_2$ NPs could be magnetically separated and partially regenerated after the decoloration. The implication for the applications of $Fe_3O_4$@$SiO_2$ NPs in water treatment is discussed.

## Introduction

Water pollution is one of the most concerned environmental problems that limit the development of human society nowadays.[1] Organic pollutants cause serious pollution to water environment, including dyes, antibiotics, pesticides, oil and other small organic molecules. To decontaminate the pollution, many technologies, such as advanced oxidation process (AOP), adsorption, activated sludge method and electrolysis, are developed and applied.[2-5] Of particular interest and importance is the AOP method, which produces highly active radicals to oxidize the electron-enriched organic pollutants.[4,5] AOP can handle diverse pollutants, especially for those not suitable for activated sludge, so AOP has been widely adopted in laboratory and industry.

Fenton reaction is the most studied and used AOP method, where $H_2O_2$ is decomposed under the catalysis of $Fe^{2+}$.[6,7] Unfortunately, $Fe^{2+}$-$H_2O_2$ system has several drawbacks that hinder its applications. The stoichiometric amounts of iron are added to the system, which consequently are oxidized into $Fe^{3+}$ ions and require further treatment to get removed. The optimal condition for the production of hydroxyl radicals is pH 3, thus large quantities of acid are essential and a neutralization step is required afterward.

To these regards, heterogeneous Fenton-like catalysts based on iron or other metals are extensively researched to overcome the aforementioned drawbacks of $Fe^{2+}$-$H_2O_2$ system.[8-15] Many studies have demonstrated that under sonication or irradiation, $Fe_3O_4$ nanoparticles (NPs) could decontaminate many organic pollutants.[16,17] Very recently, we showed that upon coating $Fe_3O_4$ NPs with a thin layer of carbon, $Fe_3O_4$ NPs catalyzed the Fenton-like reaction effectively without external energy supply.[18] However, the carbon coated $Fe_3O_4$ NPs only worked well in acidic environment. Therefore, designing suitable core-shell structure of $Fe_3O_4$ NPs as Fenton-like catalyst in neutral environment becomes the major challenge.

In this study, we reported the preparation of silica coated $Fe_3O_4$ NPs ($Fe_3O_4$@$SiO_2$ NPs) as high-performance Fenton-like catalyst in neutral environment for the decoloration of methylene blue (MB). Silica was deposited on $Fe_3O_4$ NPs by the hydrolysis of tetraethyl orthosilicate (TEOS). The decoloration efficiency of MB was measured. Influencing factors were investigated to optimize the parameters, where $Fe_3O_4$@$SiO_2$ NPs showed even higher performance at neutral pH than under acidic conditions. The magnetic separation and regeneration were also performed. The implication to the applications of $Fe_3O_4$@$SiO_2$ NPs as Fenton-like catalyst in water treatment is discussed.

## Experimental

### Materials

TEOS was bought from Jinshan Chemical Reagent Co., Ltd, China. $FeCl_2$ was purchased from Damao Chemical Reagent Co., Ltd, China. $FeCl_3$ was bought from Bodi Chemical Engineering Co., Ltd, China. MB was obtained from Sinopharm Chemical Reagent Co., Ltd, China. 5,5-dimethyl-1-pyrroline-N-oxide (DMPO) was purchased from Sigma Co., America. They were used without further purification. Other chemicals were of analytical grade.

### Preparation of $Fe_3O_4@SiO_2$ NPs

$FeCl_2\cdot 4H_2O$ (0.131 g) and $FeCl_3\cdot 6H_2O$ (0.269 g) were added to 50 mL water and the pH was adjusted to 12 under vigorous stirring. The mixture was further stirred for 1 h to allow the full precipitation. After the co-precipitation, $Fe_3O_4$ NPs were collected and washed with deionized water for three times. The coating of $SiO_2$ layer was achieved by the hydrolysis of TEOS in the presence of $NH_3\cdot H_2O$. Briefly, $Fe_3O_4$ NPs (0.1 g) were added to 1 mL of water, and then 10 mL of isopropanol was added. The mixture was sonicated for 10 min before the addition of TEOS. The mass ratios of $Fe_3O_4$ NPs: TEOS were in the range of 10:1~1:10. The mixture was shaken at 100 rpm under 35 ˚C for 5 h. The obtained $Fe_3O_4@SiO_2$ NPs were washed by water for three times and alcohol twice. The final product was dried under vacuum overnight.

Transmission electron microscopy (TEM, JEM-200CX, JEOL, Japan), X-ray photoelectron spectroscopy (XPS, Kratos, UK), Brunauer-Emmett-Teller (BET) technique (ASAP2010, Micromeritics, USA), infrared spectrometer (IR, Magna-IR 750, Nicolet, USA) and magnetometer (MPMS XL-7Tesla, Quautum Design, USA) were adopted to characterize $Fe_3O_4@SiO_2$ NPs.

### Decoloration of MB

To decolorize MB, 20 mL of MB (pH 6.5, 50 mg/L) was incubated with 20 mg of $Fe_3O_4@SiO_2$ NPs for 2 h (100 rpm at 35 ˚C) on a thermostat shaker (CHA-S, Jintan Hankang Electronic Co., China), during which the adsorption of MB reached the equilibrium. Before the addition of $H_2O_2$, 50 μL of the supernatant was collected for the absorbance ($A_0$) measurements (664 nm) on a UV-Vis spectrometer (UV1800, PGeneral, China). Then, 1.5 mL of $H_2O_2$ was added to the mixture and incubated. At each time interval, 50 μL of the supernatant was collected for absorbance ($A_t$) measurements. The decoloration efficiency was calculated as $(1- A_t/A_0) \times 100\%$. The decoloration efficiency of $Fe_3O_4@SiO_2$ NPs of different compositions was also measured. For comparison, the activity of $Fe_3O_4$ NPs was evaluated following the same protocol. As a control, the protocol was performed without adding catalyst. The chemical oxygen demand (COD) was determined using Hach reagent (low range 3~150 mg/L) on Hach DR900. The kinetic constant $k$ of decoloration was calculated following equation 1.

$$-\ln(C/C_0) = kt \qquad (1)$$

### Influencing factors

To investigate the influence of $H_2O_2$, 20 mg of $Fe_3O_4@SiO_2$ NPs was used to decolorize 20 mL of MB (pH6.5, 50 mg/L) at 35 ˚C in the presence of different volumes of $H_2O_2$.

To investigate the influence of catalyst amount, $Fe_3O_4@SiO_2$ NPs of different amounts and 1.5 mL of $H_2O_2$ were used to decolorize 20 mL of MB (50 mg/L) of different pH values (3.5-8.5) at 35 ˚C.

To investigate the influence of pH, 20 mg of $Fe_3O_4@SiO_2$ NPs and 1.5 mL of $H_2O_2$ were used to decolorize 20 mL of MB (50 mg/L) of different pH values (3.5-8.5) at 35 ˚C. Similarly, the influence of pH on the performance of $Fe_3O_4$ core was investigated.

To investigate the influence of temperature, 20 mg of $Fe_3O_4@SiO_2$ NPs and 1.5 mL of $H_2O_2$ were used to decolorize MB (20 mL, pH6.5, 50 mg/L) at different temperature (0-45 ˚C).

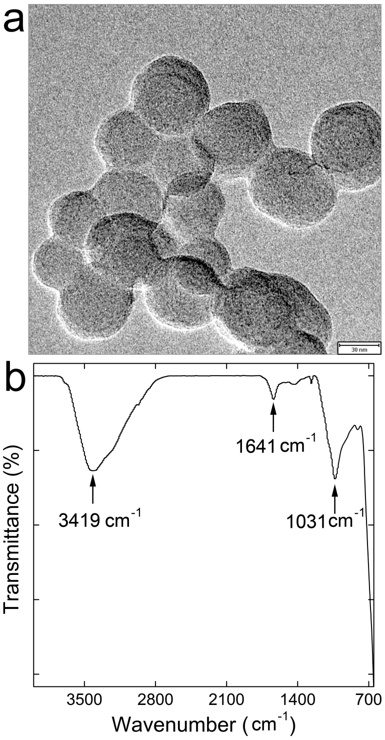

**Fig. 1** TEM (A) and IR (B) spectrum of $Fe_3O_4@SiO_2$ NPs.

To investigate the influence of inhibitor, 20 mg of $Fe_3O_4@SiO_2$ NPs and 1.5 mL of $H_2O_2$ were used to decolorize 20 mL of MB (pH6.5, 50 mg/L) at 35 ˚C in the presence of different amounts of tertiary butanol.

### Magnetic separation and regeneration

To recycle $Fe_3O_4@SiO_2$ NPs, the used $Fe_3O_4@SiO_2$ NPs were magnetically separated with a magnet. The separated $Fe_3O_4@SiO_2$ NPs were washed with deionized water by stirring for 20 min. After washing for three times, the recycled $Fe_3O_4@SiO_2$ NPs were dried and the catalytic activity was determined following aforementioned protocol. The procedure was repeated to reach the cycles of 6. Separately, the used $Fe_3O_4@SiO_2$ NPs were reduced by vitamin C (10 mg/mL) for 2 h. After washing, the reduced $Fe_3O_4@SiO_2$ NPs were subjected to activity measurement as aforementioned.

### Electron spin resonance (ESR) assay

For ESR assay, 100 μL sample was collected from the reaction solution ($Fe_3O_4@SiO_2$ NPs/$H_2O_2$ system) at 5 min and immediately mixed with 20 μL of 0.2 mol L$^{-1}$ DMPO to form DMPO-·OH adduct. The ESR spectrum was obtained on a JEOLJES FA200 facility with microwave bridge (receiver gain: $1\times10^5$; modulation amplitude: 2 Gauss; microwave power: 4 mW; modulation frequency: 100 kHz).

## Results and discussion

### Characterization of $Fe_3O_4@SiO_2$ NPs

$Fe_3O_4@SiO_2$ NPs were spherical particles with diameters of 20-40 nm. The core-shell structure could be roughly distinguished in Fig. 1a, where the resolution was limited by the magnetic interference to reach a clear verification of the core-shell

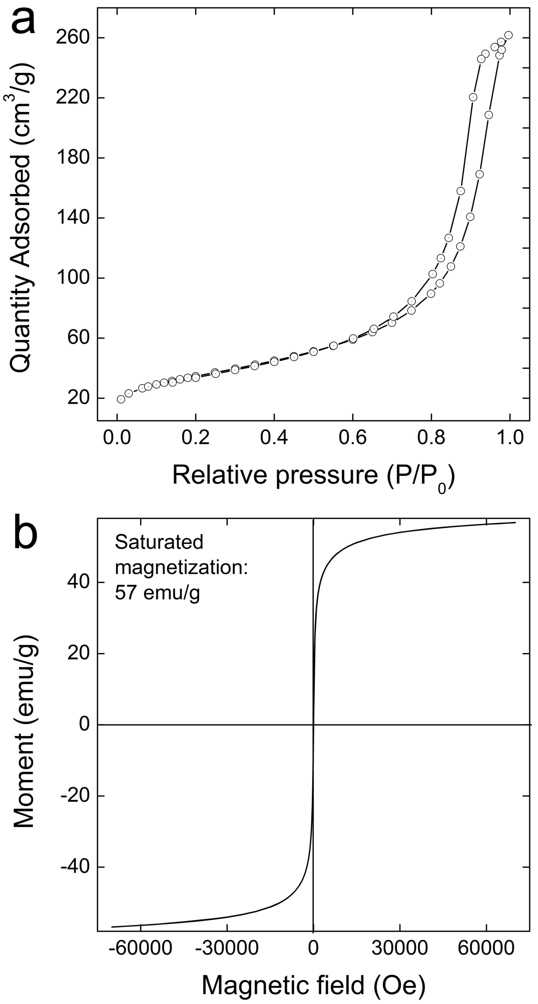

**Fig. 2** Nitrogen adsorption isotherm (A) and magnetic hysteresis loop (B) of $Fe_3O_4@SiO_2$ NPs.

structure. The IR spectrum in Fig. 1b was similar to those of typical $SiO_2$ materials. The peak at 3419 cm$^{-1}$ was attributed to the antisymmetric stretching vibration of -OH. The peak at 1641 cm$^{-1}$ was attributed to the bending vibration of -OH. The peak at 1031 cm$^{-1}$ was attributed to the antisymmetric stretching vibration of -O-Si. The chemical components were further analyzed by XPS. There were 24.5 wt% of Si and 34.6wt% of Fe in $Fe_3O_4@SiO_2$ NPs.

The BET measurement indicated that the specific surface area of $Fe_3O_4@SiO_2$ NPs was 124.3 m$^2$/g, and the total pore volume was 0.405 cm$^3$/g. The surface area of $Fe_3O_4@SiO_2$ NPs was larger than that of $Fe_3O_4$ core (112.7 m$^2$/g). The large surface area and the amorphous $SiO_2$ would benefit the pre-concentration of pollutants around $Fe_3O_4@SiO_2$ NPs, because amorphous $SiO_2$ is good adsorbent for many pollutants. The $N_2$ adsorption/desorption isotherm curve followed type IV (Fig. 2a), suggesting the nature of adsorption hysteresis. The magnetic property measurement found that the saturated magnetization was 57 emu/g, weaker than that of bulk $Fe_3O_4$ (92.8 emu/g),[19] which could be attributed to the small size of NPs.[20] The magnetic hysteresis loop indicated that $Fe_3O_4@SiO_2$ NPs was ferromagnetic (Fig. 2b). The outstanding magnetic property allowed the magnetic separation of $Fe_3O_4@SiO_2$ NPs after the water treatment.

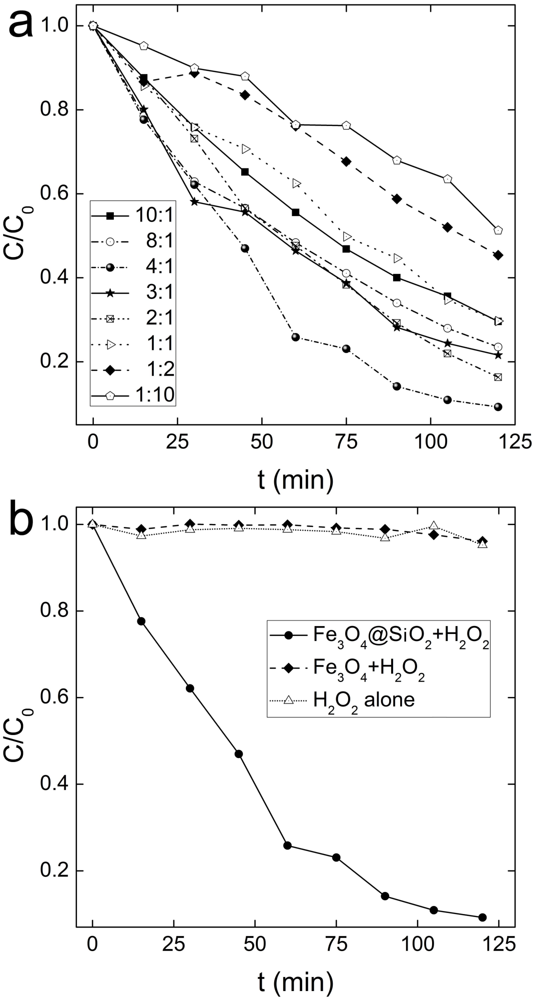

**Fig. 3** Decoloration of MB in $Fe_3O_4@SiO_2$-$H_2O_2$ system at neutral pH. (A) the effect of initial $Fe_3O_4$:TEOS mass ratio on the decoloration; (B) the comparison between $Fe_3O_4@SiO_2$ NPs and bare $Fe_3O_4$ cores.

### Catalytic activity of $Fe_3O_4@SiO_2$ NPs

The catalytic activity of $Fe_3O_4@SiO_2$ NPs was reflected by the decoloration capability of MB. We optimized the parameters of the preparation protocol. As shown in Fig. 3a, different initial ratios of TEOS: $Fe_3O_4$ led to different catalytic efficiency. The optimal ratio was 1:4, with a decoloration efficiency of 91%. The corresponding COD decreased from 101 mg/L to 25 mg/L after decoloration. The kinetic constant *k* at this ratio was calculated as 0.020 min$^{-1}$ (Figure S1). This was competitive to those of the high-performance nanocatalysts in the literature.[21-23] For example, Hsieh et al. reported the *k* value of FePt NPs-$H_2O_2$-MB system was 0.0033~0.023 min$^{-1}$ at pH 5.5.[21] When more TEOS was added, the decoloration efficiency decreased. A possible explanation could be that more TEOS induced a compact coating of $Fe_3O_4$ core, which hindered the diffusion of MB toward the $Fe_3O_4$ surface. Beyond that, too much $SiO_2$ might also block the electron-transfer from iron to MB. On the other hand, when less TEOS was added, the pre-concentration effect of $SiO_2$ shell was not dominating. Correspondingly, the promotion in catalytic activity was less.

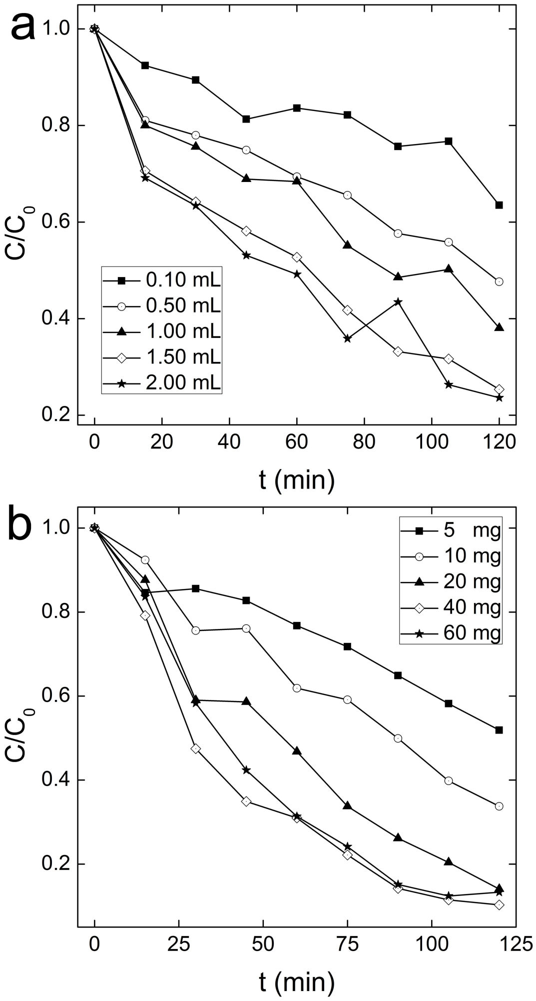

**Fig. 4** Influence of $H_2O_2$ (A) and $Fe_3O_4@SiO_2$ NPs (B) on the decoloration of MB in $Fe_3O_4@SiO_2$-$H_2O_2$ system.

Nevertheless, coating $SiO_2$ was very effective in enhancing the catalytic activity of $Fe_3O_4$. Although bare $Fe_3O_4$ cores had similar specific surface area, bare $Fe_3O_4$ NPs had no catalytic activity in the decomposition of $H_2O_2$ at near neutral pH (Fig. 3b). The kinetic constant $k$ was calculated as 0.00020 $min^{-1}$, suggesting the much slower decoloration kinetics (Figure S1). Bare $Fe_3O_4$ NPs only worked at pH 3.5 ($k$=0.0046 $min^{-1}$) and 8.5 ($k$=0.0041 $min^{-1}$) in our experiments (Figure S2), where the kinetics were still much lower than that of $Fe_3O_4@SiO_2$-$H_2O_2$ system. The comparison clearly indicated that $SiO_2$ coating was vital for the high performance of $Fe_3O_4@SiO_2$ NPs. $SiO_2$ at least had two important effects on $Fe_3O_4@SiO_2$ NPs. Firstly, amorphous $SiO_2$ could adsorb MB, which led to the pre-concentration of MB around $Fe_3O_4$ cores.[24,25] When radicals were generated, more radicals could reach MB before the self-extinction. Another effect might be that $SiO_2$ facilitated the electron-transfer from iron to MB.[26-28] Collectively, these effects resulted in the high catalytic activity of $Fe_3O_4@SiO_2$ NPs in neutral environment.

## Influencing factors

We investigated the influencing factors for $Fe_3O_4@SiO_2$ NPs. As indicated in Fig. 4a, more $H_2O_2$ was favorable in promoting the decoloration efficiency. Adding 1.5 mL and 2.0 mL of $H_2O_2$ showed the highest decoloration efficiency. To save $H_2O_2$, we recommended 1.5 mL $H_2O_2$. More $Fe_3O_4@SiO_2$ NPs also benefited the decoloration (Fig. 4b). 20 mg of $Fe_3O_4@SiO_2$ NPs showed competitive activity to 40 and 60 mg. Thus, 20 mg was used through our experiments. It was reasonable that more $H_2O_2$ and $Fe_3O_4@SiO_2$ NPs promoted the decoloration from the views of kinetics and equilibrium.

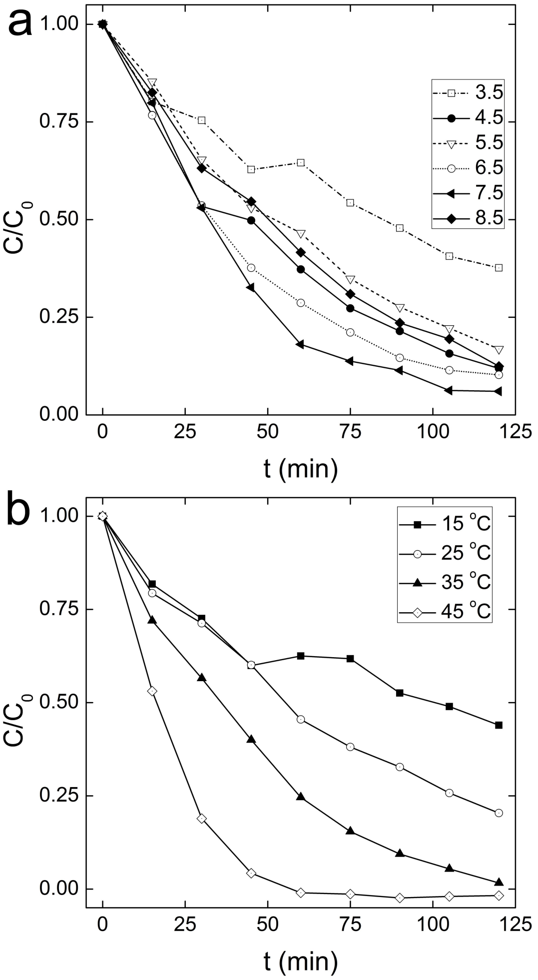

**Fig. 5** Influence of pH (A) and temperature (B) on the decoloration of MB in $Fe_3O_4@SiO_2$-$H_2O_2$ system.

In the evaluations of pH, $Fe_3O_4@SiO_2$ NPs showed very high activity at near neutral pH values (Fig. 5a). From pH 4.5 to pH 8.5, the decoloration efficiencies were all over 80%. At pH 6.5 the decoloration achieved 90% and the ratio was 94% at pH 7.5. The results indicated an important merit of $Fe_3O_4@SiO_2$ NPs that $Fe_3O_4@SiO_2$ NPs could be used in neutral environment without external energy supply.[23] It was somehow surprising that $Fe_3O_4@SiO_2$ NPs showed less activity at pH 3.5, which was the optimal pH for traditional $Fe^{2+}$-$H_2O_2$ system. This might imply that the fundamental mechanism of the catalysis was changed. Similar phenomena were reported in literature, too.[29,30] However, the mechanism of such changes still requires future investigations.

In the evaluations of temperature, the decoloration performance of $Fe_3O_4@SiO_2$ NPs showed a temperature-dependent manner (Fig. 5b). Obviously, higher temperature benefited the decoloration. This was consistent with our previous observation of $Fe_3O_4@C$ NPs, where higher temperature facilitated the diffusion of MB and generation of

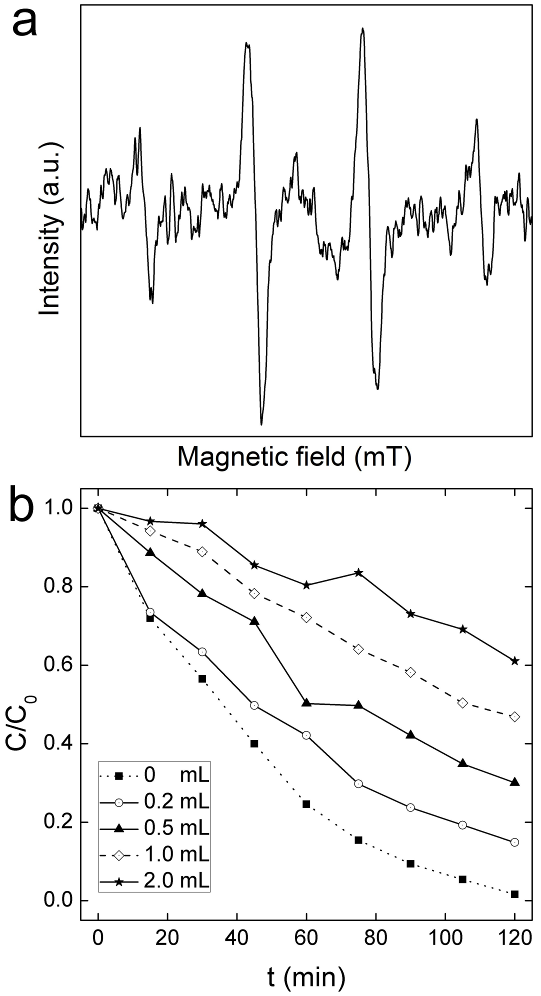

**Fig. 6** (A) DMPO spin-trapping ESR spectrum of ·OH radicals in $Fe_3O_4@SiO_2$-$H_2O_2$ system; (B) influence of tertiary butanol on the decoloration of MB in $Fe_3O_4@SiO_2$-$H_2O_2$ system.

radicals.[18] It should be noted that the decoloration efficiencies at 35 °C and 45 °C were very close, although the decoloration efficiency reached the maximum much faster.

The presence of radical scavenger (tertiary butanol) inhibited the decoloration. Mechanistically, the decoloration was achieved by the attack of radicals generating during the decomposition of $H_2O_2$. As shown in Fig. 6a, the ESR spectrum confirmed the presence of ·OH radicals upon the catalysis of $Fe_3O_4@SiO_2$ NPs. The ESR spectrum in the presence of $Fe_3O_4@SiO_2$ NPs displayed a 4-fold characteristic peak of the typical DMPO-·OH adduct with an intensity ratio of 1:2:2:1. When the radicals were eliminated before they reached the pollutants, the decontamination would be blocked. To this regard, we suggested that radical scavengers should be avoided during the decontamination. Again, $SiO_2$ coating was much better than C coating.[18] In our previous report, only 100 μL of tertiary butanol could completely inhibit the decoloration. In this study, the performance of $Fe_3O_4@SiO_2$ NPs was only inhibited by 13%. Even when 2 mL tertiary butanol was added, the decoloration efficiency was retained as 39%. A possible explanation could be that the affinity of MB to $Fe_3O_4@SiO_2$ NPs was stronger than that of tertiary butanol, which resulted in the accumulation of MB rather than tertiary butanol around $Fe_3O_4@SiO_2$ NPs. Therefore, the radicals reached MB before the scavenging by tertiary butanol. Further investigations on the mechanism are highly encouraged.

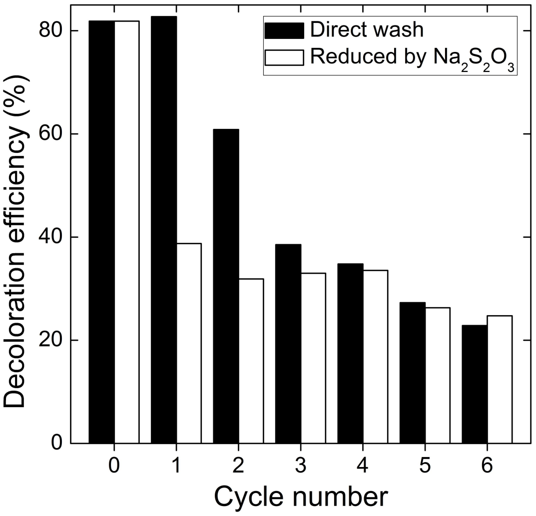

**Fig. 7** Recycling of $Fe_3O_4@SiO_2$ NPs after the decoloration of MB.

### Separation and regeneration

After the decoloration, $Fe_3O_4@SiO_2$ NPs could be magnetically separated with an external magnet. The used $Fe_3O_4@SiO_2$ NPs were washed with deionized water to remove most remnant MB. The regenerated $Fe_3O_4@SiO_2$ NPs was used directly after the wash or dried for storage. The catalytic activity of recycled $Fe_3O_4@SiO_2$ NPs was determined (Fig. 7). At cycle one, the catalytic activity of $Fe_3O_4@SiO_2$ NPs was well retained without obvious loss. Thereafter, the recycling led to moderate decrease of activity of $Fe_3O_4@SiO_2$ NPs. The decrease became slow when the cycle number reached 3. Despite the decrease of catalytic activity, the results indicated that $Fe_3O_4@SiO_2$ NPs could be partially regenerated after the Fenton-like reaction. Thus, $Fe_3O_4@SiO_2$ NPs were more environment-friendly than traditional $Fe^{2+}$-$H_2O_2$ system.

There might be a possibility that $Fe_3O_4$ core was oxidized during the treatment and the oxidation of $Fe_3O_4$ resulted in the activity loss. To exclude this, we reduced the recycled $Fe_3O_4@SiO_2$ NPs with $Na_2S_2O_3$ (Fig. 7). The reduction did not improve the regeneration, implied that the oxidation of $Fe_3O_4$ was not the main reason of the activity loss. Similar phenomena were observed when vitamin C and hydrazine hydrate were used as the reducer (data not shown). Herein, we speculated that the loss of catalytic activity might be due to the destruction of some vulnerable domains of $Fe_3O_4@SiO_2$ NPs. After 3 cycles, the relative stable sites survived and the catalytic activity became almost constant.

## Conclusions

In summary, $Fe_3O_4@SiO_2$ NPs was prepared as high-performance Fenton-like catalyst for the decoloration of MB, where neutral pH was the optimal condition. $SiO_2$ coating is crucial in promoting the catalytic activity and shifting the optimal pH toward neutral. Higher temperature was preferred and the radical scavengers should be avoided in the applications of $Fe_3O_4@SiO_2$ NPs. The magnetic separation of $Fe_3O_4@SiO_2$ NPs after the decoloration made the process environment-friendly. We hope that our results will stimulate more interests on the applications of high-performance nanocatalysts for the environmental remediation.

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## Notes and references

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† Electronic Supplementary Information (ESI) available: decloloration kinetics constants; catalytic performance of $Fe_3O_4$ core under different pH values. See DOI: 10.1039/b000000x/

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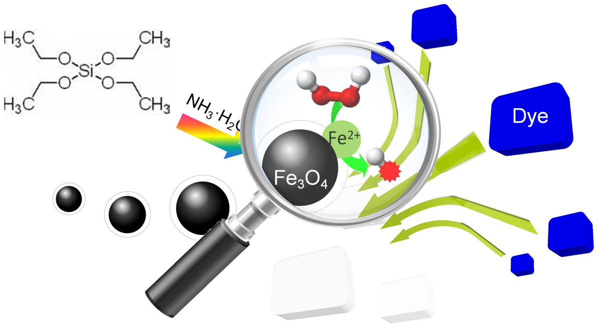

$Fe_3O_4@SiO_2$ could be used as high-performance Fenton-like catalyst for dye decoloration in neutral environment.